

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
Reply to Office Action of: January 13, 2005

Claims 1 – 42 remain in this application. Claims 22-23 have been amended to overcome the section 112 rejection, where the unbleachable loss of claim 23 having the antecedent basis from claim 22 is not even mentioned anymore in claim 23 and also amended for better clarity. Claims 1-21 and 24-42 have been withdrawn as a result of an earlier restriction requirement. In view of the examiner's earlier restriction requirement, applicant retains the right to present claims 1-21 and 24-42 in a divisional application.

### **1. § 103 Rejections**

The Examiner has rejected claims 22-23 under 35 U.S.C. § 103(a) as being unpatentable for obviousness over Fleming (U.S. Patent No. 4,872,895), Campion (U.S. Patent No. 5,888,587), or a combination of the two and optionally in view of DiGiovanni (U.S. Patent No. 5,123,940).

The Examiner agrees that Flemming and Camion do not explicitly teach some of the specifics of the processing, such as the amount of loss, the viscosity, the ratio etc. The Examiner asserted it would have been obvious to have the smallest loss possible or to perform routine experimentation to determine optimal amount of dopant in the glass.

However, the optimal amount of dopant for the answer to one problem is not necessarily the same amount of dopant for the answer to a different problem, which is the case here. Contrary to the Examiner's assertions that specifics of the processing would be obvious, the motivation to even arrive at Applicants' process or its benefits are not even suggested by the references, alone or in combination.

The substance of the Applicant's invention is that one can use even a very coarse batch material and obtain a level of homogeneity that is comparable to that obtained via a much more difficult and expensive sol-gel process, as taught in the references. The word "batch" for those in the field, means a mixture of raw materials, each of which has a different nature (e.g., particle size, or size distribution) and/or chemistry (e.g., SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub>). Unlike the sol-gel approach of the references, there is no limit to the

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
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compositional diversity that can be obtained from the final result of the Applicants' claimed process. There is also no need to resort to exotic melting strategies such as a soot gun to obtain the final melted glass.

The one attribute that the claimed invention has over the references is the use of the starting material of the fiber-making process with natural occurring silica and not just synthetic silica, as is more clearly described in amended claim 23.

In general, the phrase "starting silica source" refers to the raw material used to make a silica source, e.g., octamethylcyclotetrasilane (OMCTS) used for the flame hydrolysis process. The starting silica source, such as OMCTS, is not the same as, for example, a "source of silica," which would be silicon dioxide and nothing more.

Previous prior art showed that rare earth doping could be achieved by dipping a preform in a solution of a dissolved rare earth salt. The preform however was a CVD made blank which has a high porosity before consolidation into a glass body. To those technically knowledgeable in the field, the word "preform" refers to a solid body, porous or otherwise, and not to the raw materials per se. So the CVD process requires a synthetic silica blank or body first. The references cited by the Examiner teach the process for making a sol-gel derived glass which again is a process incorporating a synthetically prepared silica, not sand or the more general claimed "macroscopic silica powder substrate" of claim 22.

The main benefit to the Applicants' invention is that natural occurring silica or sand can be used, as a powder substrate, to form a glass having a good distribution of optically active ions in the glass body. The ability to distribute the optically active ion in a glass matrix provides the benefit for allowing a low unbleachable loss due to minimizing intra ionic cross relaxation. Theoretically, one could melt a glass having an optically active ion and let it sit at low viscosity (high temperatures) for a period of time to allow the ions to move around and create equilibrium in the glass to minimize cross relaxation. However, this period of time could be on the order of days or weeks. The problem is that at high temperatures the glass solution volatilizes certain glass constituents and the composition will change and especially on the surface of the glass creating a non-homogeneous glass mixture. This is detrimental to producing good homogeneous glass

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
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for fiber applications. The claimed invention allows a good distribution of the optically active ion without incurring the long melting times, which is a problem the references did not solve, and hence can not obviate.

Referenced patents Fleming (4872895) (3954431) and Campion (5888587) are basically stating that using a Sol-Gel method, a precursor soot can be fabricated and by shooting this soot through a plasma torch it can be melted and formed into a glass. Any dopant mentioned in the patents are based on a need to change the index of the glass or to improve mechanical strength (Campion column 2 line 17). Reference patent DiGiovanni (5123940) is discussing the use of a sol-gel technique to coat a tube again to give an index change in a resulting fiber. Hence, the references are not using dopants to solve the same problem of unbleachable loss as the Applicants.

In general, to use the sol-gel or CVD processes one usually have to have silica in some kind of liquid starting state (not Applicants' powder) which means a silane such as tetraethoxysilane, silicatetrachloride, trimethoxysilane etc. So the starting silica source is a compound containing Silicon. When the solution is mixed with water or alcohol solvents one get Si-X where X could be OH and/or organic groups can chemically bonded. When heat treated usually by flame the compound breaks down to form O-Si-O networks of bonds i.e. a glass.

In contrast, the Applicants' invention starts with a solid (powder) naturally occurring Si-O<sub>2</sub> (quartz, cristobalite, tridymite) source with no solvents and coat this silica source with a solution of a dissolved rare earth salt in a solvent (i.e. water only) (this is a solvent having no silicon present and is not the same solvent referred to in the sol-gel process by the Examiner) and coat the surface of the individual SiO<sub>2</sub> grains with the rare earth solution.

For example, the Applicants' invention teaches a method of coating the surface of solid particles of silicon dioxide (quartz, synthetic silica, Cristobalite, tridymite) with a solution of a dissolved optically active ion element in a solvent. Adding dopants does not reduce the relaxation by itself. However, by the act of coating the surface of a macroscopic silica particle with a solution containing the dopant, an improved (lower) cross relaxation behavior results by increasing the distribution of the ions.

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
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Here is a crude way of looking at the Applicants' process in simplistic terms. Think of making pancakes and the need to add an egg to the pancake mix. The egg represents the dopant, the mix the silica. One does not just drop the egg into the mix and then throw it on the griddle. If just such a simple drop is done, then one would basically have an egg cooked into the pancake (i.e. poor ion distribution with a high intra-ionic cross relaxation, therefore high unbleachable loss). Instead, if the egg is stirred or otherwise "mixed" into the pancake mix and allowed to coat the mix particles, the egg evenly distributes its beneficial properties to the mix and makes a good pancake (i.e. low intra-ionic cross relaxation, wide distribution of ions creating a low unbleachable loss in the fiber amplifier). In terms of the glass melting if one just takes a rare earth oxide (egg not broken up) and add it to the batch (pancake mix), the ions do not distribute as well as if one took a rare earth salt and make a solution (beaten egg) and allow it to coat the batch or specifically the macroscopic silica for the solution to distribute the ion more evenly by stirring or otherwise mixing.

Whereas the coated silicon dioxide is insoluble in the solvent, the silicon dioxide plus optically active ion solution is heat treated to drive off excess solvent and mixed with other raw materials making up the glass composition. The mixture of raw materials is then heat treated at temperatures that allow a viscosity of the resulting melted material to be below 10,000 poise. The resulting glass can then be drawn into a fiber.

Assuming the Examiner argues that dipping a CVD blank in a rare earth solution suggests the Applicants' invention for a synthetic silica, the naturally occurring silicon dioxide (quartz, cristobalite, tridymite) that could be used as the Applicants' starting material still can not be used in the conventional dipping CVD process.

The Applicants' invention is intended to provide a technique to dope a glass with an active rare-earth ion which can be subsequently drawn into a fiber. An active ion is one that as energy strikes the ion its electrons can be excited into a different energy level. Then after a period of time the electron relax and fall back down to a lower energy level and while doing this gives energy. This energy that is given off is used to help amplify a signal that is being transmitted through a fiber. Previously withdrawn Claims #14 and claim #15 list the possible optically active ions. None of the referenced patents talk about

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
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optically active ions. Rare-earth elements are preferred because most transition elements are contaminants in a fiber amplifier and need to be down in the ppt levels to make a good fiber. Some co-doping of fibers with a rare earth elements and aluminum have been done with the teachings of the Applicants' invention, as examples.

A problem with doping a fiber with a rare earth element and using standard techniques to fabricate the glass such as vapor deposition or traditional melting is that if the optically active ions are not separated from neighboring ions, energy that is given off by the electron relaxing to a lower energy level can be absorbed by a neighboring ion. If the neighboring ion absorbs the energy then this distracts from the amplification efficiencies of the fiber. So the optimum fiber would have enough optically active ions spaced sufficiently apart for minimal cross relaxation and maximum amplification (gain). The Examiners comments about "obvious to mix everything enough so that everything is uniform" is true but just mixing does not provide the dispersal of the optically active ions enough to give the unbleachable loss achieved by the solution doping of the sand.

All the patent references are using a sol-gel technique to make a precursor soot which is not the process in the claimed invention. The limitation in amended claim 22 of "macroscopic" means sand-sized (10s to 100s of microns, discernable to the naked eye, even if very small), as opposed to microscopic, which would be micron-sized (a few microns or less, too small to make out as individual grains by eye). The word "macroscopic" is used to help differentiate between sand and CVD soot (where soot is usually on the order of sub-micron particles).

The sol-gel technique uses a solution usually made up of metal alkoxides or organo-metallic compounds of the required elements which are dissolved in an alcoholic or aqueous solution. By adjusting the pH of the solution the constituents can polymerize to form a gel which can be dried or sintered into a powder. This powder or solution can be doped with other elements in the form of salts that can be incorporated in the gel and soot. Usually the soot is formed from very fine particles and has to be melted together either by passing the soot through a flame or plasma. The melting of the soot is usually done in microseconds and the resulting glass particles are captured on a bait rod or substrate. The

Appl. No.: 10/686,862  
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resulting material is considered synthetic silica and not naturally occurring silica such as sand for the Applicants' process.

In contrast, the claimed invention can use natural occurring silica (sand) or synthetic silica which is coated with the optically active ion. One can go to the beach and take a cup of sand and use it as the claimed invention teaches to make a doped glass but can not do this with the referenced processes. Conventionally, one does not use beach sand due to impurities but the present invention shows any silica source is viable. To reduce the cross relaxation of the optically active ions, coating the individual sand particles with a solution of the dissolved rare earth salt provides the means to distribute the ions through out the glass.

Another difference is that in the Applicants' invention, one heat treats the sand coated with the rare earth ion at lower temperatures in which the dopant is not chemically bonded to the silica but merely attached to the outer surface of the sand particle. When the coated sand particle is melted along with other raw materials at elevated temperatures that is when the ion is incorporated into the glass. In the reference patents, the dopants are chemically part of the gel or soot formed through the sol-gel process.

The Examiner commented on water as the solvent with reference to Campion column 2 lines 17-23 and column 1 lines 55-57. The water is the solvent in the sol-gel process in which a soluble solvent is added to achieve chemically bonded elements prior to creating a gel. The references have a solution containing a silica precursor such as (TEOS tetra ethyl ortho silica) an organic silica compound that can chemically bond with the dopant.

In contrast, the claimed invention uses water as a solvent to dissolve the rare earth element salt to coat a silica particle and not create a chemically bonded compound. As a matter of fact, the silica particles are chosen to be insoluble in the solvent to allow the silica particles to be coated with the dissolved rare earth element.

Mixing is already mentioned but is the basis for the claimed invention. By just adding a rare earth containing salt to a glass batch, as discovered by the Applicants, a surprising result was discovered that the optically active ions were sufficiently dispersed enough to provide good fiber performance.

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
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The melting difference is that in the Applicants' invention, the rare earth ion has to be incorporated into the glass matrix by melting the raw materials together to a sufficient viscosity that allows the optically active ion to become part of the glass network.

In contrast, the references are melting a sol-gel derived powder or soot having the dopant already part of the network and just creating a glassy material by using a torch or plasma. Again the timing of the melting activity with a plasma is micro-seconds compared to the Applicants' process of melting the raw materials for several hours or more.

Another benefit of the Applicants' claimed process that is not present in the sol-gel and CVD type glass fabrication processes is Applicants' diversity possibilities. In the conventional sol-gel and CVD type glass fabrication processes, there are limitations to the compositions that can be made. With the Applicants' invention, any glass composition of interest can be prepared through coating the sand particles with a dopant and adding the sand with other raw materials and melting the raw materials together. For example, the glass that was prepared for erbium fiber amplifiers at Corning had a high amount of antimony in the glass. This glass could not be prepared by a CVD or a sol-gel process.

The whole discussion on viscosity is a moot point and Examiner is respectfully requested to determine the viscosity of the material shot through a torch or plasma. The Examiner's reference to lead and tin is not understood as to relevance. The claimed invention's reference to viscosity is based on the forming of a glass by thermally melting the batch materials which can also include antimony oxide and alumina along with the coated silica.

In summary, the Applicants are using optically active ions from rare earth elements to create a glass having the property of amplification of energy signal by the up conversion of the rare earth ion. The sol-gel process is used to make glass but is not related to the claimed invention which is using raw materials that are not chemically bonded prior to melting the glass. The main difference of the claimed invention lies with the start of an unobvious raw material. In general, "raw material" refers to the chemicals

Appl. No.: 10/686,862  
Amdt. Dated: April 5, 2005  
Reply to Office Action of: January 13, 2005

which, subjected to some sort of transformative process, convert to the final homogeneous material desired. For example, OMCTS is a raw material used in a flame hydrolysis process to make silica soot, tetraethylorthosilicate is a raw material used to make a silica preform via sol-gel, and sand is the raw material silica source used in the batch for a melted silicate glass only taught by the Applicants' claimed invention and hence unobviated by the references, alone or in combination.

Based upon the above amendments, remarks, and papers of records, applicant believes the pending claims of the above-captioned application are in allowable form and patentable over the prior art of record. Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Applicant believes that no extension of time is necessary to make this Reply timely. Should applicant be in error, applicant respectfully requests that the Office grant such time extension pursuant to 37 C.F.R. § 1.136(a) as necessary to make this Reply timely, and hereby authorizes the Office to charge any necessary fee or surcharge with respect to said time extension to the deposit account of the undersigned firm of attorneys, Deposit Account 03-3325.

Please direct any questions or comments to Juliana Agon at (607) 974-6574.

April 5, 2005  
Date

<p><b>CERTIFICATE OF TRANSMISSION</b> <b>UNDER 37 C.F.R. § 1.8</b></p> <p>I hereby certify that this paper and any papers referred to herein are being transmitted by facsimile to the U.S. Patent and Trademark Office at 703-872-9306 on:</p> <p><u>April 5, 2005</u> Date</p> <p><u>Juliana Agon</u> 4/5/05 Juliana Agon Date</p>
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